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MASTER

TITLE: THIN-FILM THICKNESS MEASUREMENT USING X-RAY PEAK RATIOING IN THE SCANNING ELECTRON MICROSCOPE

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THIN FILM THICKNESS MEASUREMENT USING X-RAY PEAK RATIOING IN THE SCANNING ELECTRON MICROSCOPE

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Measurement of sub-micrometer films on substrates has been a problem arising in association with a variety of vacuum deposition techniques. In particular, laser fusion targets have provided a focus for attention because the films are often opaque, and because of the small, spherical surfaces of targets and the stringent requirements for symmetry. The Scanning Electron Microscope (SEM) with its finely focused probe and ability to both image and analyze a specimen provides an ideal environment for the examination of such targets.

The specimens we have looked at so far have been glass microballoons metallized with gold. The principle for measuring thin film thickness with the SEM is shown in Figure 1. The electron beam from the instrument impinges on the sample, penetrates the gold film, and is stopped in the glass substrate, generating both gold and silicon x-rays. The ratio of the integrated counts in these two peaks is proportional to the film thickness as shown in Figure 2.

Our technique overcomes problems inherent in other schemes using the electron excited x-ray signal to measure film thickness. Bolon and Lifshin[1] used a similar scheme except they ratioed to the gold signal from a pure, bulk gold sample. We could not use this technique because of electron beam instabilities intrinsic to the field emission electron

gun used in our SEM. More generally applicable theoretical techniques[2,3] have likewise proven difficult to apply because of beam instability or limitations on the film thickness which can be measured.

The procedure we have used to obtain film thickness is summarized as follows: An x-ray spectrum is acquired from the specimen to an approximate standard deviation (1) of 1.5% for the net intensity of the largest peak in the spectrum. X-ray background is then removed from the entire spectrum. At this point a correction is applied to the SiKα peak to account for the presence of the interfering AuM_z peak which is unresolved by both energy-dispersive and wavelength dispersive x-ray detectors. This correction is determined from the x-ray spectrum of a bulk gold specimen and amounts to approximately 5% of the AuMα,β net intensity for our system. The ratio of the gold to silicon net counts is calculated and converted to a thickness using the calibration curve in Figure 2. The empirical nature of the technique overcomes many instrument-operator sources of error. However, certain conditions must be controlled to achieve adequate results. Most important is that the x-ray takeoff angle used during determination of the calibration curve be repeated during sample measurement. The takeoff angle is defined as the angle between the central ray of the cone of x-rays intercepted by the detector and the plane of the specimen surface. The high mass absorption coefficient of gold for silicon x-rays will give erroneous results if the absorption path length changes from standard to specimen. Obviously, attention must also be given to duplicating composition and density between standards and the specimen to be measured.

Applying this technique to targets produced in varying deposition geometries we have estimated the measurement to have a accuracy of approximately 5% and a precision of approximately 3%. The useful thickness range for gold is 100 to 2000Å in our instrument using a 20kV supply.

This method is generally applicable to any coating on any substrate as long as the electron energy is sufficient to penetrate the coating and the substrate produces an x-ray signal which can pass back through the coating and be detected.

REFERENCES

1. R. B. Bolon and E. Lifshin in SEM/1973 Proceeding of the 6th Annual SEM Symposium (O. Johari, Ed.) IITRI, Chicago, Illinois (1973), p. 285.

2. W. E. Sweeney, R. E. Seebold, L. S. Birks, J. Appl. Phys., 31, 1061 (1961).

3. H. Yakowitz and D. E. Newbury in SEM/1976 Proceedings of the 9th Annual SEM Symposium (O. Johari, Ed.), IITRI, Chicago, Illinois (1976), p. 151.

FIGURE CAPTIONS

Figure 1. Schematic Representation of the Electron Beam-Specimen Interaction Showing the Source of X-Ray Signals Used in Thin Film Measurement.

Figure 2. Gold Film Calibration Curve for Glass Substrate. E_0 is the Accelerating Potential of the Electron Gun.

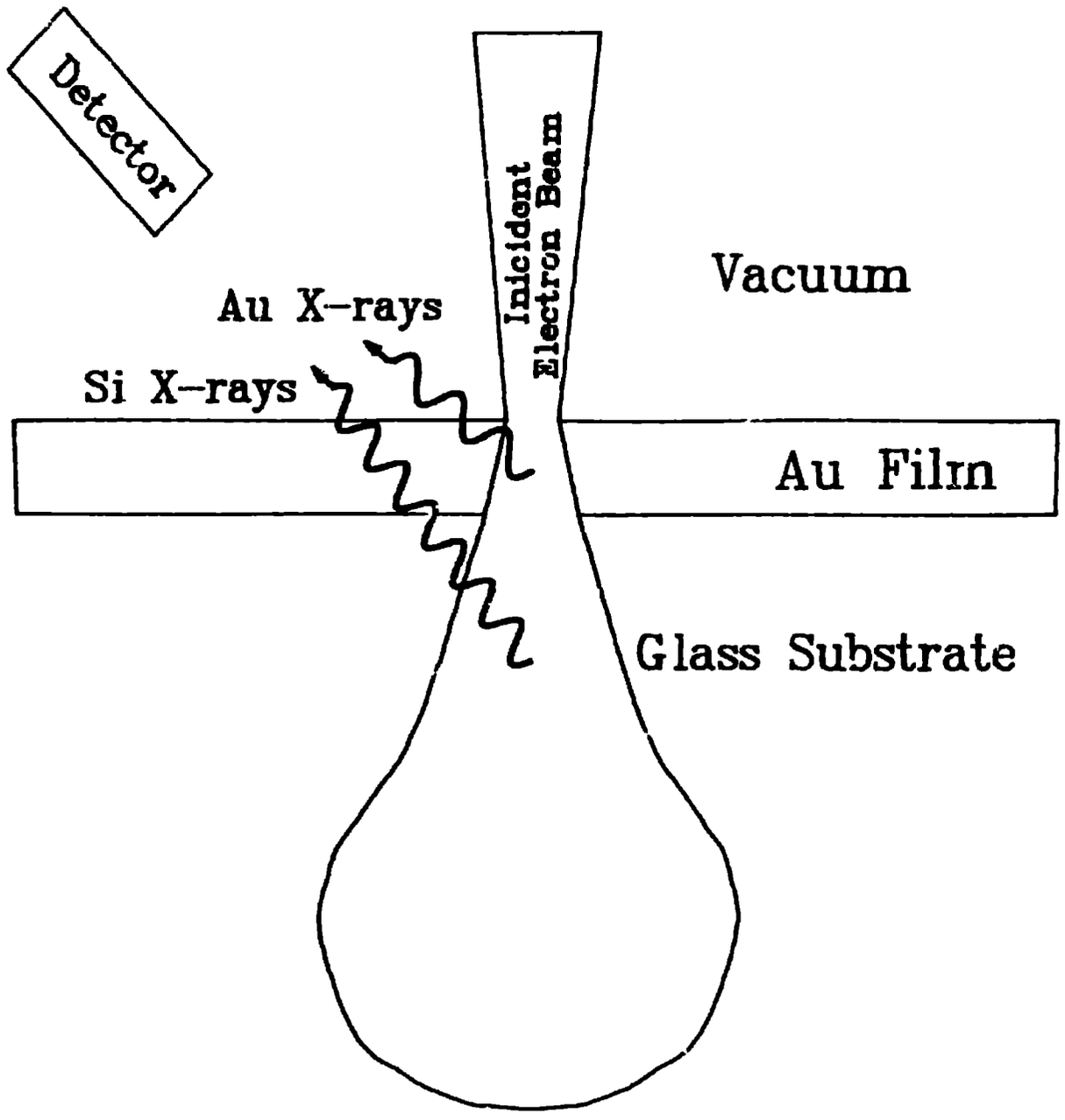
Detector
Incident Electron Beam
Vacuum
Au X-rays
Si X-rays
Au Film
Glass Substrate

Figure 1

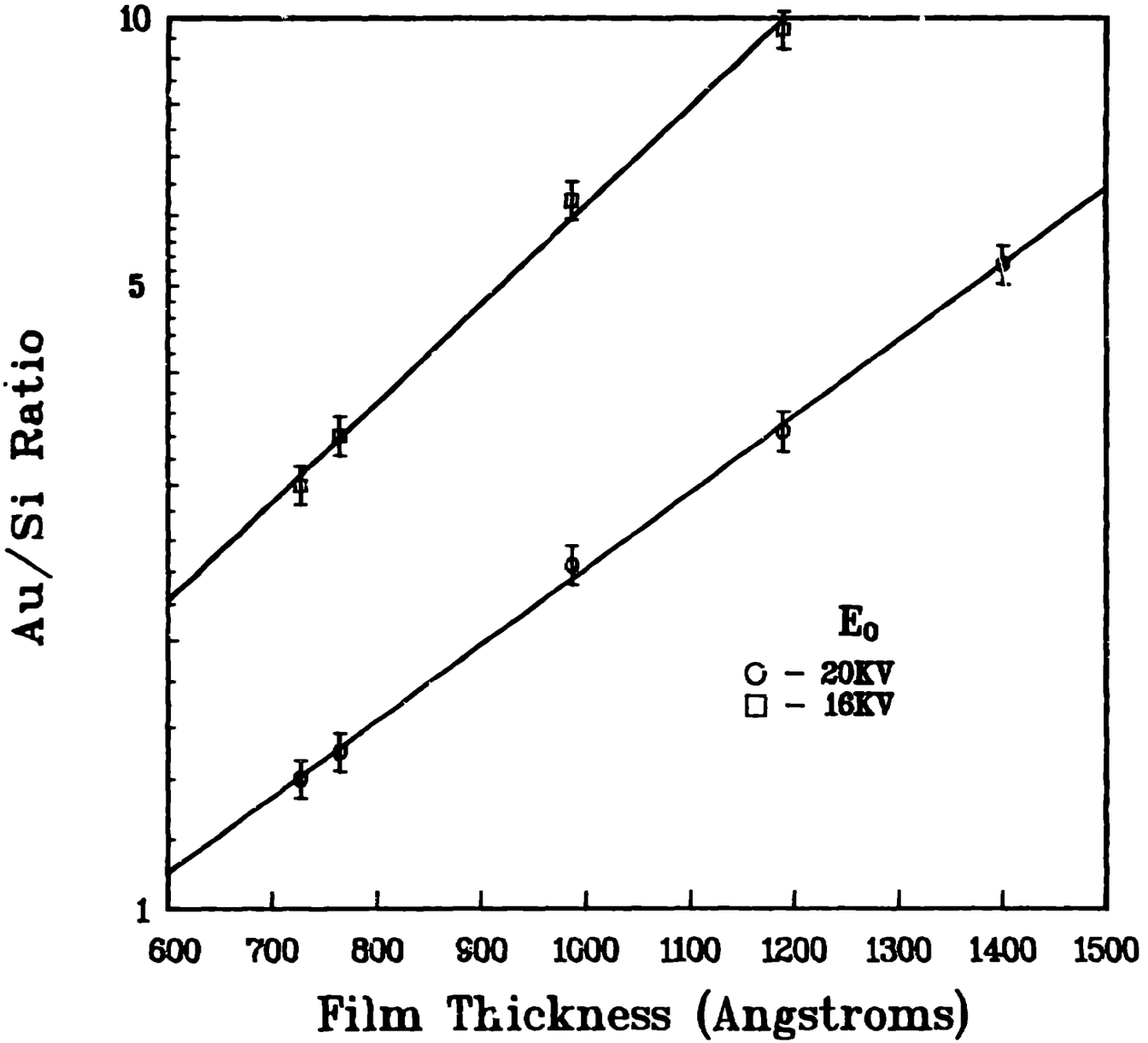
10
5
1
Au/Si Ratio
600
700
800
900
1000
1100
1200
1300
1400
1500
Film Thickness (Angstroms)
E0
○ – 20KV
□ – 16KV

Figure 2